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(54) DUST CORE FOR HIGH FREQUENCY AND MANUFACTURING METHOD THEREFOR**(57)Abstract:**

PROBLEM TO BE SOLVED: To solve the problem where breakage and exfoliation are generated in an insulating covering layer and desired characteristic is not obtained, when a core is molded in high density in order to correspond to miniaturization of a device by improving characteristics of the dust core, which is formed by molding ferromagnetic metal powder whose surface is covered with an insulating material to realize high magnetic flux density, high permeability, low iron loss and high strength, so as to obtain a dust core superior in both magnetic characteristics and strength.

SOLUTION: In a mixed powder, an inorganic insulating material and an organic insulating material which serves as binder are mixed in ferromagnetic metal powder by a volume ratio that the total of this materials is 1-6% (in which ratio of the inorganic insulating material is 0.5-5.5%). The mixed powder is subjected to fusing treatment, where strong compressing and shearing action is applied mechanically and repeatedly. Since obtained covering layers are bonded stiffly to inner powder, breakage and exfoliation are not generated when high pressure molding is performed, and a dust core superior in both magnetic characteristics and strength is obtained.

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CLAIMS

[Claim(s)]

[Claim 1] The dust core for RFs characterized by being the enveloping layer which presents the organization condition united with the surface section of ferromagnetic metal powder after this enveloping layer contained the both sides of an inorganic insulating material and an organic insulating material and both had distributed minutely in the dust core which consists of ferromagnetic metal powder with which the firm insulating enveloping layer was formed in the front face.

[Claim 2] The dust core for RFs according to claim 1 a total of 1 - 6% (among those, an inorganic insulating material 0.5 - 5.5%) and whose ferromagnetic metal powder the organic insulating materials of ferromagnetic metal powder and an insulating material which serve both as an inorganic insulating material and a binder by the volume ratio comparatively are the remainders.

[Claim 3] The manufacture approach of the dust core for RFs characterize by press this powder into a necessary configuration and carry out heating solidification after perform fusion processing which carry out the repetitive load of the powerful compression / shear operation to the mixed powder which blended with ferromagnetic metal powder the organic insulating material which serve both as an inorganic insulating material and a binder mechanically and form a firm insulating enveloping layer in the front face of ferromagnetic metal powder .

[Claim 4] the manufacture approach of the dust core for RFs characterize by press this powder into a necessary configuration and carry out heating solidification after perform fusion processing which carry out the repetitive load of the powerful compression / shear operation to the mixed powder which carried out combination of the organic insulating material which serve both as an inorganic insulating material and a binder to ferromagnetic metal powder by the volume ratio a total of 1 to 6 % (among those, an inorganic insulating material 0.5 - 5.5 %) mechanically and form a firm insulating enveloping layer in the front face of ferromagnetic metal powder.

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DETAILED DESCRIPTION

[Detailed Description of the Invention]

[0001]

[Field of the Invention] This invention relates to the dust core used for various kinds of electrical and electric equipment, and the suitable flux density for cores, such as a choke coil, a noise filter, and a reactor, especially used in the high frequency field to 1kHz - about 1MHz is highly excellent in frequency characteristics, and it is related with the dust core which can respond to the miniaturization of a device, for example.

[0002]

[Description of the Prior Art] In the iron core (core) of the soft magnetic materials used in an alternating current magnetic field, it is required that flux density and permeability are large and that iron loss should be especially small. Although silicon steel suits this condition, when a configuration is complicated, it calls at the core (powder core) by the powder metallurgy which can be fabricated in the configuration of arbitration in many cases, in order to receive constraint in a configuration by the relation which carries out the laminating of the punching article of sheet metal, and makes it. Although the so-called sintering iron core which sintered the green compact, and the dust core which solidified the powder of ferromagnetic metals, such as pure iron, a Fe-Si alloy, Sendust, and a permalloy, with thermosetting resin, water glass, and other proper binding material, without sintering are located in a powder core, since the eddy current loss which occupies a part for the principal part of iron loss is proportional to the square of the thickness of an iron core, it has the problem that iron loss becomes large in the sintering iron core of one shaping.

[0003] At this point, in the case of a dust core, since nonmagnetic resin intervenes among iron powder children, there is the essential description that eddy current loss is small. Since flux density becomes settled uniquely with the density ratio of a core in the case of a dust core, if the rest is fabricated to high density and flux density is raised, it can satisfy demand characteristics. In order to raise the consistency of a green compact, it is necessary to reduce making high compacting pressure at the time of pressing powder, and the frictional resistance produced between powder and between powder and metal mold in connection with it, and, generally powder lubricant is mixed into raw material powder as the means. However, although frictional resistance is reduced in the case of the dust core which does not have a sintering process depending on the amount, the fall of green density may be caused on the contrary, or the powder lubricant fused in process of resin hardening may bar junction of iron powder and resin and good formation of a resin layer, and, as a result, the fall of the reinforcement (anti-****) of a green compact may be caused. Therefore, in the case of a dust core, it is desirable to stop addition of powder lubricant as a little as possible, and to use force-plunger lubrication together.

[0004]

[Problem(s) to be Solved by the Invention] This applicant sets to JP,49-15684,B before. In a powder core as well as the case of the iron system magnetism alloy by the usual solution process, addition of a component with the specific resistance of each component single tastes, such as Si, aluminum, and nickel, or the specific resistance of the solid solution with iron higher than the specific resistance of ***** is effective in improvement in magnetic properties to iron.; especially Improving [if it is made the organization in which the thin layer of these components or the diffusion section of those covers an iron grain child, and exists / alternating current magnetic properties (flux density, iron loss)]-remarkably;, and such organization Since the iron powder by which the front face was covered with silicone was obtained when immersing and drying iron powder in the water solution of silicone when Si was taken for the example for example, it indicated realizing

easily by use of the iron powder.

[0005] As a result of also miniaturizing the core used for these as the miniaturization of various electrical and electric equipment progressed in recent years, even if it miniaturized, the conventional function was not spoiled, i.e., it has magnetic properties, such as high flux density, quantity permeability, and low iron loss, and the core of high intensity came [however,] to be searched for. However, as a result of the friction wear between powder increasing if the addition of powder lubricant is reduced and compacting pressure is made high in order to carry out powder compacting of this to high density even if it uses the powder in which the layer with high specific resistance was formed on the front face, in conventional mere granulation or conventional above-mentioned immersion processing extent, the enveloping layer of **** exfoliated and an expected property was not reached. Then, the technical problem of this invention is to find [much more quality-of-the-material-amelioration of an enveloping layer, and] out a means to combine that enveloping layer with the front face of ferromagnetic metal powder (ferromagnetic metal powder is represented with iron powder on these specifications below.), such as iron powder, firmly.

[0006]

[Means for Solving the Problem] An artificer first variously as a result of research about the quality of the material of an enveloping layer If the enveloping layer of the organization condition united with an iron powder child's surface section after the enveloping layer contained the both sides of an inorganic insulating material and an organic insulating material and these both had distributed minutely is formed, desired magnetic properties will be acquired.; In that case About the blending ratio of coal of iron powder and a cladding material (an inorganic insulating material and organic insulating material) Especially the presentation range of 1 - 6% (among those, an inorganic insulating material 0.5 - 5.5%) and the remaining iron powder has the desirable sum of an inorganic insulating material and an organic insulating material at a volume ratio.; although the enveloping layer of such an organization condition is not obtained with the above-mentioned common use means of the passage former It found out being obtained easily, when performing processing which carries out the repetitive load of the powerful compression / shear operation to the mixed powder which blended iron powder and insulating material powder with the predetermined rate mechanically.

[0007] There is equipment called the mechanical particle compound-ized equipment of a compression shearing die as equipment suitable for this processing, and it is supposed that it will be applicable to production of a covering form composite particle, the surface treatment of a particle, configuration control, promotion of fusion between solid particulates, precision mixing, etc. There are a mechano fusion (surface fusion) system of Hosokawa Micron CORP., a hybridization system of the Nara machine factory, and the theta composer (all are the trade names) and others of TOKUJU in a commercial item, and each principle is similar. If the thing of first in a roll is taken for an example, equipment will consist of arm material with the rotating container and the circular head with which it was equipped into it, and it is pushed against a container inside, rotates with a container, and the supplied fine particles receive powerful compression / shear operation between a head and a container inside, adhere to a container inside, and are scratched by the centrifugal force with a scraper. These are repeated at high speed and effectiveness, such as formation of particle compound, shows up. Although the clearance between a container inside and a head is adjusted according to the class and the processing purpose of processed powder, it is about 50-500 micrometers in general.

[0008] According to the test result of an X diffraction and others, although the enveloping layer which makes an insulating material a subject on that front face by using iron powder as a nucleus will be formed if this processing is performed to the mixed powder of iron powder and insulating material powder, a nuclear metal phase and the insulating material particle made detailed distribute by turns, and this enveloping layer serves as an organization made amorphous the part, and shows the insulation of very high resistance. And it turns out that both have united in an interface since it is changing continuously with inclination negative in one [inclination forward / distribution / of both component / concentration / in one component, and] of other components near the interface of a nuclear metal phase and an insulating material particle. Thus, since the enveloping layer and the nucleus are unifying firmly, even if it presses this by the high-pressure force, unlike the case of the conventional approach of granulation, immersion, and others, the case in the end of composite powder this processing was performed produces destruction and exfoliation of an enveloping layer, and does not cause degradation of a property. Incidentally, suppose that it is called "fusion processing" on these specifications as a name suitable to this processing. The meaning also has the processing which similarly means covering with a

mere granulation and the mere conventional compound in aiming at distinction with these, since the effectiveness of covering completely differs.

[0009] The mechanism in which the enveloping layer firmly united with the nucleus by the above-mentioned organization is formed is considered as follows by performing this fusion processing to the mixed powder of iron powder and insulating material powder. That is, minutely, the insulating material powder inserted into iron powder (nuclear particle) is ground and divided by powerful compression / shear operation received in case mixed powder passes through between the container inside of a processor, and heads, and adheres on the surface of a nuclear particle according to it. On the front face of a nuclear particle, the adhering insulating material powder is embedded by compressive force, and the mixed phase (enveloping layer) of a nuclear metal phase and the insulating material particle made detailed is formed gradually. And in the interface of a nuclear metal phase and an insulating material particle, with the frictional heat produced between nuclear particles, both react partially and fix (fusion). The enveloping layer equipped with the desired description is obtained as a result of this repeat.

[0010] As ferromagnetic metal powder, it excels in soft magnetic characteristics, and the high iron system metal powder of saturation magnetic flux density is desirable. Pure iron powder is suitable in price especially, and although the iron powder by the atomizing method and the various manufacturing methods of reduction and others is used, the point of compression-molding nature or purity to reduced iron powder is desirable. Incidentally the purity of reduced iron powder is usually more than 99.9 mass %. Although a Fe-Si system, a Fe-Si-aluminum system, a Fe-nickel system, a Fe-Co system, a Fe-Mo-nickel system, etc. are mentioned as ferromagnetic metal powder other than pure iron, according to flux density, desired permeability, and desired cost, it is chosen suitably. About the particle size of ferromagnetic metal powder, eddy current loss becomes small, its RF property improves so that particle size is small, but if too small, a powdered fluidity and compression-molding nature will worsen and the dust core of high density will not be acquired. Therefore, about 10-100 micrometers 150 micrometers or less are preferably suitable for particle size.

[0011] as an inorganic insulating material -- aluminum $2O_3$, SiO_2 , TiO_2 , and $CaCO_3$ etc. -- mineral product powder, such as oxide powder and a kaolin (kaolin), diatomaceous earth, and talc (talc), is used. The smaller possible one of the particle size of an inorganic insulating material is desirable in order to obtain a detailed mixing phase, and its 50 micrometers or less are desirable. However, since there is a problem of handling or cost when it passes minutely, it is good to be referred to as 0.5 micrometers or more. Thermoplastics powder, such as thermosetting resin powder, such as a phenol, epoxy, and polyimide, and a polyamide, polyethylene, polyphenylene sulfide, is used for an organic insulating material. About the particle size of an organic insulating material, 1-50 micrometers is desirable at the reason same with having described the inorganic insulating material.

[0012] Although the synthetic resin as an organic insulating material is an indispensable component which serves as the binder of a dust core, if independent, sufficient insulation is difficult to get, and in order [this] to secure especially the insulation in a RF field, it is necessary to use it together with an inorganic insulating material. And the presentation range of 1 - 6% (among those, an inorganic insulating material 0.5 - 5.5%) and the remaining iron powder has [especially the loadings to ferromagnetic metal powder (iron powder)] the sum of an inorganic insulating material and an organic insulating material desirable at a volume ratio in that case. As the data of the example mentioned later show the reason, it is because an inorganic insulating material cannot secure the insulation of a request of the sum of an inorganic insulating material and an organic insulating material in a RF field at less than 1%, and reinforcement of a dust core less than 0.5%, but flux density and permeability will fall remarkably if an inorganic insulating material exceeds 5.5% on the other hand and the sum of an inorganic insulating material and an organic insulating material becomes superfluous exceeding 6%, and the reinforcement of a core also falls to coincidence. In addition, unless it refuses, the volume ratio has shown altogether the blending ratio of coal in this specification, especially an addition, etc.

[0013]

[Embodiment of the Invention] About raw material powder, into the pure iron powder as a representative of ferromagnetic metal powder, first The particle size of 100 micrometers or less (It describes like -100 micrometers hereafter.) Although phosphoric-acid coat processing was performed to reduced iron powder and this iron powder, two kinds insulating material powder For an inorganic insulating material, talc powder (-3 micrometers), alumina powder (-5 micrometers), Two kinds, phenol resin powder (-50 micrometers) and

polyphenylene-sulfide-resin (PPS;-30micrometer) powder, were prepared for the organic insulating material for four kinds of silica powder (-3 micrometers) and calcium-carbonate powder (-30 micrometers). Organic resin serves as the binding material in a dust core. Subsequently, it blended with the rate of a law everywhere which showed such raw material powder in Table 1, and the mixed powder a sample 1 - for sample 23 with which presentations differ, respectively was prepared. It is shown that * mark of the column of resin (organic) is polyphenylene sulfide resin (the-less mark is phenol resin) about front Naka and * mark of the column of iron powder being iron powder which performed phosphoric-acid coat processing.

[0014] next, in order to see the operation effectiveness of the fusion processing by which it is characterized [of this invention], other samples except samples 20-23 add and mix 0.05% (each sample -- uniform) of powder of ethylene-bis-stearamide as powder lubricant, after performing fusion processing to each mixed powder using a theta composer (trade name of TOKUJU). on the other hand, about samples 20-23, 0.05% (each sample -- uniform) of powder of ethylene-bis-stearamide is added and mixed as a non-processed example of a comparison, without performing fusion processing. Subsequently, although it is the translation which produces the core for magnetic-properties measurement, and the test piece for measurement on the strength for every sample, in order to see the effect of whenever [stoving temperature / which solidifies the compacting pressure and the green compact at the time of powder compacting], these factors are assigned as shown in each of each sample and Table 1.

[0015] The core for magnetic-properties measurement has the shape of a ring with the bore of 20mm, an outer diameter [of 30mm], and a thickness of 5mm. Moreover, the test piece for measurement on the strength is plate-like [with die length of 31.8mm, a width of face / of 12.7mm /, and a thickness of 5mm], and evaluates reinforcement with the anti-****. The alcoholic suspension of ethylene-bis-stearamide is beforehand applied and dried as a die lubricant at the metal mold which fabricates these at every shaping. Heating time for the solidification after shaping is set as for 30 minutes at 350 degrees C, when whenever [stoving temperature] is 180 degrees C, and it is set as for 20 minutes at 500 degrees C for 1 hour.

[0016] (Example) According to the conditions first shown in the column of the sample 9 of Table 1, into the reduced iron powder which performed phosphoric-acid coat processing, as an inorganic insulating material 4.5% of talc (talc) powder, Blend 0.5% of powder of phenol resin as an organic insulating material which serves as binding material, and fusion processing is performed by the theta composer. Powder lubricant was added 0.05%, after fabricating in a configuration predetermined by compacting pressure 980MPa, heating of 1 hour was performed at the temperature of 180 degrees C, and the dust core and the anti-**** test piece were produced. The dust core and anti-**** test piece which are applied to a sample 1 - a sample 8 and a sample 10 - a sample 23 like the following according to each combination presentation and processing conditions which are shown in Table 1 were produced, and the characteristic test was presented. In addition, a theta composer's service conditions are 500 micrometers of clearances between a container inside and a head, and both 2.5m [/second] rotational-speed difference, and the processing time is for 30 minutes.

[0017] Next, about the trial of magnetic properties, alternating current magnetic properties gave the coil of 20 primary coils and 20 secondary coils to the core, measured effective-permeability μ_{ea} in the frequency of 1kHz, 400kHz, and 1MHz, and asked for the ratio (in a table, it is written as 1M/1k) to the effective permeability in 1kHz of the effective permeability in 1MHz. This ratio is one of the measure which evaluates a RF property, and it is shown that the effective permeability of the core in a desired frequency range is stable, i.e., the RF property is excellent, so that this value is close to 1. Direct-current magnetic properties give the coil of 200 primary coils and 20 secondary coils to a core, and are flux density B100. It measured. Moreover, anti-**** laid the test piece in the material testing machine in the distance between the supporting points of 25.4mm, set it as the core for the load, and asked for disruptive strength.

[0018] Thus, the magnetic properties of the obtained sample 1 - a sample 23 and the data of anti-**** are shown in Table 2. Originally, since Table 1 and this table 2 are what divided one table on account of space, in order to make it legible, in the existence of the outline of the combination column of Table 1 and fusion processing of the remarks column of Table 2 in Table 2, they overlap Table 1, respectively and have been carried to it. The array of the sample in a table is what performed fusion processing which makes samples 1-19 the main point of this invention altogether, and it has arranged in order with few insulating material loadings. As the data of Table 2 show, since permeability decreases sharply as a frequency becomes high from 1kHz to 1MHz, a sample 1 - a sample 3 cannot be used for the core in this RF field. Since this has the insufficient

insulating material content at less than 1%, it is considered to be because for an enveloping layer with necessary insulation not to be formed.

[0019] On the other hand, since there are many insulating material contents as 6% ** (superfluous), although formation of an insulating enveloping layer is enough and a RF property is excellent, since the rates of the iron powder in a core run short on the other hand, a sample 17 - a sample 19 are inferior in the effective permeability, flux density, and anti-****, and do not reach desired quality. On the other hand, although the insulating material of especially a desirable thing is 5% (95% of iron powder), the sample 4 which has an insulating material in 1 - 6% of within the limits - the sample 16 fulfill the quality of a request of magnetic properties and reinforcement enough. Therefore, the thing a sample 4 - a sample 16 are the examples of this invention, and a sample 1 - a sample 3 have [thing] a RF property inferior in the loadings of an insulating material (sum of an inorganic insulating material and an organic insulating material) at less than 1%; a sample 17 - a sample 19 have an insulating material as superfluous as 6% **, and are made out of range [all / this invention] by permeability, flux density, and reinforcement being inferior. The following sample 20 - a sample 23 are the so-called dust cores of a conventional type, and are the example of a comparison which excluded and pressed fusion processing, the loadings of an insulating material adopting 5 optimal% among an example, and mixing raw material powder.

[0020] Here, the examination result about other factors is explained in advance of the examination about the fusion processing by which it is characterized [of this invention]. If the sample 6 whose loadings are 4.5% equally - a sample 15 are first seen about the class of inorganic insulating material, although each property of the numeric value of a calcium carbonate is a little high, a significant difference will not be accepted as a whole. This has the same insulating material loadings also from the superfluous samples 18 and 19.

[0021] Next, about the effectiveness of the phosphoric-acid coat processing to iron powder, although it is thought in the example of a comparison which does not carry out fusion processing that it is effective once, there are data which conflict when fusion processing is performed and a sample 7 - a sample 12 are seen, and it is hard to say that it is clearly effective. The reason is considered because the effectiveness of a phosphoric-acid coat is reduced by powerful friction and compression / shear operation which are received in process of fusion processing. In addition, probably because powder properties, such as a fluidity, are improved, as for the iron powder which performed phosphoric-acid coat processing, anti-**** has become height irrespective of the existence of fusion processing.

[0022] About the compacting pressure at the time of shaping, flux density and anti-**** should mainly be influenced [the], and if the sample 6 fabricated with fact low voltage - a sample 8 and a sample 13 - a sample 15 are compared with the sample 9 fabricated with high pressure - a sample 12, flux density and its anti-**** are significant. Moreover, about anti-****, the correlation inclination of whenever [after shaping / stoving temperature] can also be seen.

[0023] About the effect of the heating (solidification) processing temperature after shaping, this heating removes distortion which is made to strengthen a green compact generally and remains to a green compact with compacting pressure. Since distortion which remains to a dust core checks the magnetic properties of structure sensitivities, such as permeability, coercive force, and iron loss (hysteresis loss), if distortion disappears with heating, its properties of these should improve. However, if the insulating component of the enveloping layer formed on the surface of iron powder is spread inside iron powder with heating on the other hand, the original insulating effectiveness will degrade magnetic properties as a disadvantage crack and a result. Although anti-**** and permeability improve so that it becomes an elevated temperature as the 180 degrees C sample 7, the 9; 350 degrees C sample 6, and the 10; 500-degree C samples 11 and 12 show whenever [stoving temperature], it is thought of because it is the operation with which the effectiveness of disappearance of distortion and advance of diffusion disagrees that a RF property is in the inclination of degradation.

[0024]

[Table 1]

試験片の作製条件

試料 No.	原料粉配合組成 (体積%)						融合 処理	成形圧 MPa	加熱 ℃	備 考
	還元鉄粉	滑石	Al ₂ O ₃	SiO ₂	CaCO ₃	樹 脂				
1	99.8	—	—	—	—	0.2	○	588	180	範囲外
2	99.3	0.2	—	—	—	0.5	○	588	180	範囲外
3	99.3	—	0.2	—	—	*0.5	○	588	350	範囲外
4	99.0	0.5	—	—	—	0.5	○	588	180	本発明
5	97.0	2.5	—	—	—	0.5	○	588	180	本発明
6	95.0	—	4.5	—	—	*0.5	○	588	350	本発明
7	95.0	4.5	—	—	—	0.5	○	588	180	本発明
8	☆ 95.0	4.5	—	—	—	0.5	○	588	180	本発明
9	☆ 95.0	4.5	—	—	—	0.5	○	980	180	本発明
10	95.0	—	4.5	—	—	*0.5	○	980	350	本発明
11	☆ 95.0	4.5	—	—	—	0.5	○	980	500	本発明
12	95.0	—	4.5	—	—	*0.5	○	980	500	本発明
13	95.0	—	—	4.5	—	*0.5	○	588	350	本発明
14	95.0	—	—	—	4.5	*0.5	○	588	350	本発明
15	95.0	—	1.5	1.5	1.5	*0.5	○	588	350	本発明
16	94.0	—	1.5	1.5	2.5	*0.5	○	588	350	本発明
17	93.0	—	2.0	2.0	2.5	*0.5	○	588	350	範囲外
18	92.5	7.0	—	—	—	0.5	○	588	180	範囲外
19	92.5	—	7.0	—	—	*0.5	○	588	350	範囲外
20	95.0	4.5	—	—	—	0.5	—	588	180	比較例
21	☆ 95.0	4.5	—	—	—	0.5	—	588	180	比較例
22	☆ 95.0	4.5	—	—	—	0.5	—	980	180	比較例
23	☆ 95.0	4.5	—	—	—	0.5	—	980	500	比較例

☆：鉄粉にリン酸被膜処理

*：ポリフェニレンサルファイド樹脂
(無印はフェノール樹脂)

[0025]

[Table 2]

磁気特性と強度の測定結果

試料 No.	配合の概要		融合 処理	実効透磁率 (μ)				磁束 密度	抗折力 MPa	備考
	還元鉄粉	樹脂		1kHz	400kHz	1MHz	1M/1k			
1	99.8	0.2	○	128	98	67	0.52	1.2	72	範囲外
2	99.3	0.5	○	120	96	76	0.63	1.2	79	範囲外
3	99.3	*0.5	○	117	93	82	0.70	1.2	85	範囲外
4	99.0	0.5	○	102	98	91	0.89	1.3	83	本発明
5	97.0	0.5	○	85	83	81	0.95	1.2	82	本発明
6	95.0	*0.5	○	75	74	71	0.95	0.9	69	本発明
7	95.0	0.5	○	74	73	71	0.96	0.9	69	本発明
8	☆ 95.0	0.5	○	74	74	73	0.99	0.9	71	本発明
9	☆ 95.0	0.5	○	80	78	77	0.96	1.1	79	本発明
10	95.0	*0.5	○	83	79	77	0.93	1.1	74	本発明
11	☆ 95.0	0.5	○	88	79	75	0.85	1.1	83	本発明
12	95.0	*0.5	○	85	80	77	0.90	1.1	81	本発明
13	95.0	*0.5	○	74	73	71	0.96	0.9	71	本発明
14	95.0	*0.5	○	79	78	77	0.97	1.1	79	本発明
15	95.0	*0.5	○	76	75	73	0.96	1.0	77	本発明
16	94.0	*0.5	○	67	67	63	0.94	0.8	65	本発明
17	93.0	*0.5	○	58	57	52	0.90	0.7	50	範囲外
18	92.5	0.5	○	52	51	50	0.96	0.7	49	範囲外
19	92.5	*0.5	○	54	52	51	0.94	0.7	45	範囲外
20	95.0	0.5	—	72	30	13	0.18	0.8	54	比較例
21	☆ 95.0	0.5	—	70	58	46	0.66	0.7	57	比較例
22	☆ 95.0	0.5	—	78	55	41	0.52	0.8	64	比較例
23	☆ 95.0	0.5	—	115	12	8	0.08	0.9	81	比較例

☆：鉄粉にリン酸被膜処理

*：ポリフェニレンサルファイド樹脂（無印はフェノール樹脂）

[0026] Even when the effectiveness of fusion processing over raw material powder looked at the sample 7 - the sample 9, and sample 11 which performed this processing at the last and those all fabricate inside or a RF property with high pressure at it by equipping magnetic properties, such as effective permeability, a RF property, and flux density, and anti-**** with quality sufficient as a dust core for RFs, it has stopped at the slight fall. Since it has joined to internal iron powder firmly, the enveloping layer to which the reason passed through fusion processing is for it to be able to be equal to the high pressure at the time of compacting, or friction wear enough. On the other hand, by the sample 20 which does not perform fusion processing - the sample 23, even when the frequency exceeded 400kHz and it fabricated with low voltage, the effective permeability decreased rapidly, and the RF property has deteriorated remarkably. Since this has the weak junction to an enveloping layer and internal iron powder, it means that exfoliation of an enveloping layer and damage arose by the pressure received at the time of shaping, or friction. And this fact shows vividly the effectiveness of the fusion processing by which it is characterized [of this invention]. In addition, it is just going to **** sensibly that anti-**** is improving along with the rise of whenever [compacting pressure or stoving temperature].

[0027]

[Effect of the Invention] Conventionally, in a RF field which exceeds 1MHz, the absolute value did not have suitable core material in the middle fields, although the ferrite core whose flux density of a low (before or after 0.4) thing is stable was used chiefly and the silicon steel laminate was used in the field to about 10kHz of numbers on the other hand. For example, although even the frequency of Sendust of 1MHz is [flux density] stable, the level cannot respond to the miniaturization of the device stated to the beginning because of extent [a little] higher than a ferrite core. However, since the effective permeability hardly falls to the frequency of 1MHz, the dust core concerning this invention is excellent in a RF property, and shows high flux density

(before or after 1.1). Therefore, this invention enables correspondence to the miniaturization of the electrical and electric equipment while expanding the application range of a dust core.

[Translation done.]

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(54) DUST CORE FOR HIGH FREQUENCY AND MANUFACTURING METHOD THEREFOR

(57)Abstract:

PROBLEM TO BE SOLVED: To solve the problem where breakage and exfoliation are generated in an insulating covering layer and desired characteristic is not obtained, when a core is molded in high density in order to correspond to miniaturization of a device by improving characteristics of the dust core, which is formed by molding ferromagnetic metal powder whose surface is covered with an insulating material to realize high magnetic flux density, high permeability, low iron loss and high strength, so as to obtain a dust core superior in both magnetic characteristics and strength.

SOLUTION: In a mixed powder, an inorganic insulating material and an organic insulating material which serves as binder are mixed in ferromagnetic metal powder by a volume ratio that the total of this materials is 1-6% (in which ratio of the inorganic insulating material is 0.5-5.5%). The mixed powder is subjected to fusing treatment, where strong compressing and shearing action is applied mechanically and repeatedly. Since obtained covering layers are bonded stiffly to inner powder, breakage and exfoliation are not generated when high pressure molding is performed, and a dust core superior in both magnetic characteristics and strength is obtained.

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[0010] As a ferromagnetic metal powder, an iron-based metal powder having excellent soft magnetic property and a high saturated magnetic flux density is desirable. Inter alia, a pure iron powder is preferable from a viewpoint of cost, an iron powder obtained by an atomizing method, a reducing method or other various manufacturing processes is used and, from a viewpoint of compression moldability and purity, a reduced iron powder is desirable. Incidentally, purity of a reduced iron powder is usually 99.9% by mass or larger. Examples of a ferromagnetic metal powder other than pure iron include Fe-Si system, Fe-Si-Al system, Fe-Ni system, Fe-Co system, and Fe-Mo-Ni system, and the powder is appropriately selected depending on desired magnetic flux density, permeability and cost. Regarding a particle diameter of a ferromagnetic metal powder, as a particle diameter is smaller, eddy current loss becomes small, and high frequency property is improved. However, when the particle diameter is too small, flowability and compression moldability of a powder are deteriorated, and a dust core of a high density is not obtained. Therefore, it is suitable that a particle diameter is 150 μm or smaller, preferably around 10 to 100 μm .

[0011] As an inorganic insulating material, an oxide powder such as Al_2O_3 , SiO_2 , TiO_2 and CaCO_3 , and a mineral powder such as kaolin (potter's clay), diatomaceous earth, and talc (talcum) are used. In order to obtain a fine mixed phase, a particle diameter of an inorganic insulating material is desirably as small as possible, preferably 50 μm or smaller. However, when the particle diameter is too fine, there is a problem of handling and

cost, therefore, the particle diameter is suitably $0.5\text{ }\mu\text{m}$ or larger. As an organic insulating material, a thermosetting resin powder such as phenol, epoxy, and polyimide, and a thermoplastic resin powder such as polyamide, polyethylene, and polyphenylene sulfide are used. A particle diameter of an organic insulating material is preferably 1 to $50\mu\text{m}$ based on the same reason as that described for an inorganic insulating material.

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(54) 【発明の名称】 高周波用圧粉磁心およびその製造方法

(57) 【要約】

【課題】 表面を絶縁物で被覆した強磁性金属粉末を成形した圧粉磁心の特性をより高磁束密度、高透磁率、低鉄損且つ高強度に向上させて機器の小型化に対応するため磁心を高密度に成形しようとする、絶縁被覆層に破損・剥離を生じて所要の特性が得られない。

【解決手段】 強磁性金属粉末に無機絶縁物と結合剤を兼ねる有機絶縁物を体積比で合計1~6% (その内無機絶縁物が0.5~5.5%) 配合した混合粉に、強力な圧縮・剪断作用を機械的に反復負荷する融合処理を施す。得られた被覆層は内部の粉末と強固に接合しているため、高圧成形しても被覆層の破損や剥離は起こらず、そのため磁気特性、強度ともに優れた圧粉磁心が得られる。

【特許請求の範囲】

【請求項1】表面に絶縁性の強固な被覆層が形成された強磁性金属粉末からなる圧粉磁心において、この被覆層が無機絶縁物と有機絶縁物の双方を含有し、且つ両者が微細に分散した状態で強磁性金属粉末の表層部と融合した組織状態を呈する被覆層であることを特徴とする高周波用圧粉磁心。

【請求項2】強磁性金属粉末と絶縁物の割合が、体積比で無機絶縁物および結合剤を兼ねる有機絶縁物が合計1～6%（その内無機絶縁物が0.5～5.5%）および強磁性金属粉末が残部である、請求項1に記載の高周波用圧粉磁心。

【請求項3】強磁性金属粉末に無機絶縁物および結合剤を兼ねる有機絶縁物を配合した混合粉に、強力な圧縮・剪断作用を機械的に反復負荷する融合処理を施して、強磁性金属粉末の表面に絶縁性の強固な被覆層を形成した後、この粉末を所要の形状に圧縮成形して加熱固化させることを特徴とする高周波用圧粉磁心の製造方法。

【請求項4】強磁性金属粉末に無機絶縁物および結合剤を兼ねる有機絶縁物を体積比で合計1～6%（その内無機絶縁物が0.5～5.5%）配合した混合粉に、強力な圧縮・剪断作用を機械的に反復負荷する融合処理を施して強磁性金属粉末の表面に絶縁性の強固な被覆層を形成した後、この粉末を所要の形状に圧縮成形して加熱固化させることを特徴とする高周波用圧粉磁心の製造方法。

【発明の詳細な説明】

【0001】

【発明の属する技術分野】この発明は各種の電気・電子機器に使用される圧粉磁心に係るもので、とくに1kHz～1MHz程度までの高周波領域で用いられる例えばチョークコイル、ノイズフィルター、リアクトルなどのコアに好適な、磁束密度が高く周波数特性に優れ、機器の小型化に対応し得る圧粉磁心に関するものである。

【0002】

【従来の技術】交流磁場内で使用される軟磁性材料の鉄心（磁心）では、磁束密度および透磁率が大きいことと、鉄損が小さいことが特に要求される。珪素鋼板はこの条件に適合するが、薄板の打ち抜き品を積層して作る関係で形状に制約を受けるため、形状が複雑な場合は、任意の形状に成形可能な粉末冶金による磁心（粉末磁心）によることが多い。粉末磁心には圧粉体を焼結した所謂焼結鉄心と、焼結せずに純鉄、Fe-Si合金、センダスト、パーマロイなど強磁性金属の粉末を熱硬化性樹脂、水ガラスその他適宜の結合材で固化させた圧粉磁心とがあるが、鉄損の主要部分を占める渦電流損は鉄心の厚さの自乗に比例するため、一体成形の焼結鉄心では鉄損が大きくなるという問題がある。

【0003】この点では、圧粉磁心の場合は鉄粉粒子の間に非磁性の樹脂が介在するために渦電流損が小さいと

いう本質的特徴がある。あとは、圧粉磁心の場合磁束密度は磁心の密度比によって一義的に定まるので、高密度に成形して磁束密度を高めれば要求特性を充足することができる訳である。圧粉体の密度を高めるためには、粉末を圧縮成形する際の成形圧力を高くすることと、それに伴って粉末相互間や粉末と金型との間に生じる摩擦抵抗を減じる必要があり、その手段として、一般的には粉末潤滑剤を原料粉に混合している。しかしその量によっては、焼結工程のない圧粉磁心の場合には摩擦抵抗は減じるものの却って圧粉密度の低下を招いたり、樹脂硬化の過程で溶融した粉末潤滑剤が鉄粉と樹脂の接合および樹脂層の良好な形成を妨げ、その結果圧粉体の強度（抗折力）の低下を招くことがある。従って圧粉磁心の場合は粉末潤滑剤の添加は出来るだけ少量に留め、押型潤滑を併用するのが好ましい。

【0004】

【発明が解決しようとする課題】以前、本件出願人は特公昭49-15684号において、通常の溶解法による鉄系磁性合金の場合と同じく粉末磁心の場合も鉄にSi、Al、Niなど各成分単味の固有抵抗、または鉄との固溶体の固有抵抗が鉄単味の固有抵抗よりも高い成分の添加が磁気特性の向上に有効であること；特に、これらの成分またはその拡散部の薄層が鉄粒子を被覆して存在する組織構造にすると交流磁気特性（磁束密度、鉄損）が著しく改善されること；そしてこの様な組織構造は、Siを例にとると例えば珪素樹脂の水溶液に鉄粉を浸漬、乾燥すれば表面が珪素樹脂で被覆された鉄粉が得られるので、その鉄粉の使用により容易に実現されることを開示した。

【0005】しかし近年、各種電気・電子機器の小型化が進むにつれてこれらに使用される磁心も小型化する必要が生じた結果、小型化しても従来の機能を損わない、即ち高磁束密度・高透磁率・低鉄損などの磁気特性を具え、且つ高強度の磁心が求められるに至った。ところが表面に固有抵抗の高い層を形成した粉末を用いても、これを高密度に圧粉するため粉末潤滑剤の添加量を減らして成形圧力を高くすると粉末相互の摩擦摩擦が増大する結果、従来の単なる造粒処理や前述の浸漬処理程度では折角の被覆層が剥離してしまい、所期の特性には達しなかった。そこでこの発明の課題は、被覆層のより一層の材質の改良と、その被覆層を鉄粉などの強磁性金属粉末（以下この明細書では鉄粉で強磁性金属粉末を代表させる。）の表面に強固に結合させる手段を見出すことにある。

【0006】

【課題を解決するための手段】発明者は種々研究の結果、先ず被覆層の材質については、被覆層が無機絶縁物と有機絶縁物の双方を含有し、この両者が微細に分散した状態で鉄粉粒子の表層部と融合した組織状態の被覆層が形成されれば所望の磁気特性が得られること；その

際、鉄粉と被覆材（無機絶縁物および有機絶縁物）の配合割合については、体積比で無機絶縁物と有機絶縁物の和が1～6%（その内無機絶縁物が0.5～5.5%）および残り鉄粉の組成範囲が特に好ましいこと；この様な組織状態の被覆層は前述の通り従来の慣用手段では得られないが、鉄粉と絶縁物粉末を所定割合に配合した混合粉に、強力な圧縮・剪断作用を機械的に反復負荷する処理を施せば容易に得られることを見出した。

【0007】この処理に適する装置としては圧縮剪断型の機械式粒子複合化装置と呼ばれる装置があり、被覆型複合粒子の作製、粒子の表面改質、形状制御、固体粒子間の融合の促進、精密混合などに応用できるとされている。市販品にはホソカワミクロン社のメカノフュージョン（表面融合）システム、奈良機械製作所のハイブリダイゼーションシステム、徳寿工作所のシートコンポーザ（何れも商品名）その他があり、原理は何れも類似している。筆頭のを例にとると、装置は、回転する容器とその中に装着された円弧状ヘッドを持つ腕部材からなり、投入された粉体は遠心力によって容器内面に押し付けられて容器とともに回転してヘッドと容器内面の間で強力な圧縮・剪断作用を受け、容器内面に付着しスクレーパーで掻き取られる。これらが高速で繰り返されて粒子複合化などの効果が出る。容器内面とヘッドとの隙間は被処理粉末の種類や処理目的に合わせて調整されるが、概ね50～500μm程度である。

【0008】この処理を鉄粉と絶縁物粉末との混合粉に施すと、鉄粉を核としてその表面に絶縁物を主体とする被覆層が形成されるが、この被覆層はX線回折その他の試験結果によれば核の金属相と微細化した絶縁物粒子が交互に分散し、一部非晶質化した組織となって極めて高抵抗の絶縁性を示している。そして核の金属相と絶縁物粒子との界面近傍では、両者の成分の濃度分布が一方の成分は正の勾配、他の一方の成分は負の勾配をもって連続的に変化していることから、界面においては両者が融合していることが判る。この様に、この処理を施した複合粉末の場合は被覆層と核とが強固に一体化しているためこれを高圧力で圧縮成形しても造粒、浸漬その他の従来の処理法の場合と異なり、被覆層の破壊や剥離を生じて特性の劣化を招くことはない。ちなみに、この処理に相応しい名称として、この明細書では“融合処理”と呼ぶこととする。その趣旨は、同じく被覆を意図する処理でも単なる造粒や従来の複合とは被覆の効果が全く異なるので、これらとの区別を図ることにある。

【0009】この融合処理を鉄粉と絶縁物粉末との混合粉に施すことにより前述の組織構造で核と強固に一体化した被覆層が形成される機序については、次のように考えられる。即ち、混合粉が処理装置の容器内面とヘッドとの間を通過する際に受ける強力な圧縮・剪断作用によって、鉄粉（核粒子）に挟まれた絶縁物粉末が微細に粉碎、分断され、核粒子の表面に付着する。核粒子の表面

上では付着した絶縁物粉末が圧縮力によって埋め込まれ、核の金属相と微細化した絶縁物粒子との混合相（被覆層）が徐々に形成される。そして核の金属相と絶縁物粒子との界面では核粒子相互間に生じる摩擦熱によって両者が部分的に反応し固着（融合）する。この繰り返しの結果、所望の特徴を具えた被覆層が得られる訳である。

【0010】強磁性金属粉末としては軟磁気特性に優れ、飽和磁束密度の高い鉄系金属粉末が望ましい。中でも価格的に純鉄粉は好適であり、アトマイズ法、還元法その他の各種製造法による鉄粉が用いられるが、圧縮成形性や純度の点から還元鉄粉が望ましい。ちなみに還元鉄粉の純度は通常99.9質量%以上である。純鉄以外の強磁性金属粉末としてはFe-Si系、Fe-Si-Al系、Fe-Ni系、Fe-Co系、Fe-Mo-Ni系なども挙げられるが、所望の磁束密度、透磁率およびコストに応じて適宜に選択される。強磁性金属粉末の粒径については、粒径が小さいほど渦電流損は小さくなり高周波特性が向上するが、小さすぎると粉末の流動性、圧縮成形性が悪くなり、高密度の圧粉磁心が得られない。従って粒径は150μm以下、好ましくは10～100μm程度が適当である。

【0011】無機絶縁物としてはAl₂O₃、SiO₂、TiO₂、CaCO₃などの酸化物粉末、およびカオリン（白陶土）、珪藻土、タルク（滑石）などの鉱産物粉末が用いられる。無機絶縁物の粒径は微細混合相を得るため出来るだけ小さい方が望ましく、50μm以下が好ましい。ただし微細に過ぎると取り扱いやコストの問題があるので、0.5μm以上とするのがよい。有機絶縁物にはフェノール、エポキシ、ポリイミドなどの熱硬化性樹脂粉末や、ポリアミド、ポリエチレン、ポリフェニレンサルファイドなどの熱可塑性樹脂粉末が使用される。有機絶縁物の粒径については、無機絶縁物について述べたのと同様の理由で1～50μmが好ましい。

【0012】有機絶縁物としての合成樹脂は、圧粉磁心の結合剤を兼ねる必須の成分であるが、これ単独では充分な絶縁性は得難く、特に高周波領域での絶縁性を確保するためには、無機絶縁物と併用する必要がある。そしてその場合、強磁性金属粉末（鉄粉）への配合量は、体積比で無機絶縁物と有機絶縁物の和が1～6%（その内無機絶縁物が0.5～5.5%）および残り鉄粉の組成範囲が特に好ましい。その理由は後述する実施例のデータが示すように、無機絶縁物が0.5%未満、無機絶縁物と有機絶縁物の和が1%未満では高周波領域での所望の絶縁性および圧粉磁心の強度が確保できず、一方、無機絶縁物が5.5%を越え、無機絶縁物と有機絶縁物の和が6%を越えて過剰になると、磁束密度および透磁率が著しく低下し、同時に磁心の強度も低下することによる。なおこの明細書における配合割合、添加量などは、特に断わらない限り全て体積比で示してある。

【0013】

【発明の実施の形態】先ず原料粉については、強磁性金属粉末の代表としての純鉄粉には粒径 $100\mu\text{m}$ 以下（以下、 $-100\mu\text{m}$ のように記す。）の還元鉄粉、およびこの鉄粉にリン酸被膜処理を施したものの二種類を、絶縁物粉末は、無機絶縁物にはタルク粉末（ $-3\mu\text{m}$ ）、アルミナ粉末（ $-5\mu\text{m}$ ）、シリカ粉末（ $-3\mu\text{m}$ ）、炭酸カルシウム粉末（ $-30\mu\text{m}$ ）の四種類を、有機絶縁物にはフェノール樹脂粉末（ $-50\mu\text{m}$ ）およびポリフェニレンサルファイド樹脂（PPS； $-30\mu\text{m}$ ）粉末の二種類を用意した。有機樹脂は、圧粉磁心における結合材を兼ねている。次いでこれらの原料粉を表1に示した各所定の割合に配合して、それぞれ組成の異なる試料1～試料23用の混合粉を調製した。表中、鉄粉の欄の☆印はリン酸被膜処理を施した鉄粉であることを、（有機）樹脂の欄の＊印はポリフェニレンサルファイド樹脂（無印はフェノール樹脂）であることを示している。

【0014】次にこの発明の特徴とする融合処理の作用効果を見るため、試料20～23を除く他の試料はそれぞれの混合粉にシータコンポーザ（徳寿工作所の商品名）を使用して融合処理を施した後、粉末潤滑剤としてエチレンビスステアロアミドの粉末0.05%（各試料一律）を添加・混合する。一方、試料20～23については無処理の比較例として、融合処理を施さずにエチレンビスステアロアミドの粉末0.05%（各試料一律）を添加・混合する。次いで各試料ごとに磁気特性測定用の磁心、および強度測定用の試験片を作製する訳であるが、圧粉時の成形圧力や圧粉体を固化する加熱温度の影響を見るため、これらの因子を各試料それぞれ、表1に示すように割り付けてある。

【0015】磁気特性測定用の磁心は内径20mm、外径30mm、厚さ5mmのリング状である。また、強度測定用の試験片は長さ31.8mm、幅12.7mm、厚さ5mmの平板状であり、その抗折力をもって強度を評価する。これらを成形する金型には成形の都度、予め押型潤滑剤としてエチレンビスステアロアミドのアルコール懸濁液を塗布・乾燥しておく。成形後の固化処理のための加熱時間は加熱温度が 180°C の場合は1時間、 350°C では30分間、 500°C では20分間とする。

【0016】（実施例）先ず表1の試料9の欄に示す条件に従い、リン酸被膜処理を施した還元鉄粉に無機絶縁物として滑石（タルク）粉末4.5%と、結合材を兼ねる有機絶縁物としてフェノール樹脂の粉末0.5%を配合してシータコンポーザにより融合処理を施し、粉末潤滑剤を0.05%添加して成形圧力980MPaで所定の形状に成形後、温度 180°C で1時間の加熱を行なって圧粉磁心と抗折力試験片を作製した。以下同様にして、表1に示すそれぞれの配合組成および処理条件に従って試料1～試料8および試料10～試料23に係る圧

粉磁心と抗折力試験片を作製し、特性試験に供した。なお、シータコンポーザの運転条件は容器内面とヘッドとの隙間 $500\mu\text{m}$ 、両者の回転速度差2.5m/秒、処理時間は30分間である。

【0017】次に磁気特性の試験については、交流磁気特性は1次コイル20回、2次コイル20回の巻線を磁心に施して周波数1kHz、400kHzおよび1MHzにおける実効透磁率 μ_a を測り、1MHzにおける実効透磁率の1kHzにおける実効透磁率に対する比（表には $1\text{M}/1\text{k}$ と略記）を求めた。この比は高周波特性を評価する物差の一つであり、この値が1に近いほど、所望の周波数範囲内における磁心の実効透磁率が安定していることを、即ち高周波特性が優れていることを示す訳である。直流磁気特性は1次コイル200回、2次コイル20回の巻線を磁心に施して磁束密度 B_{10} を測定した。また、抗折力は試験片を材料試験機に支点間距離25.4mmで載置し、その中心に負荷して破壊強度を求めた。

【0018】この様にして得られた試料1～試料23の磁気特性、抗折力のデータを表2に示す。表1とこの表2は、本来は1枚の表を紙面の都合で分割したもので、見易くするために表1には表2の備考欄を、表2には表1の配合欄の概要と融合処理の有無を、それぞれ重複して掲載してある。表における試料の配列は、試料1～19は全てこの発明の骨子とする融合処理を施したもので、絶縁物配合量の少ない順に並べてある。表2のデータが示すように、試料1～試料3は1kHzから1MHzまで周波数が高くなるにつれて透磁率が激減してしまうので、この高周波領域での磁心には使えない。これは、絶縁物含有量が1%未満で足りないために、所要の絶縁性を持つ被覆層が形成されないことによると考えられる。

【0019】一方、試料17～試料19は絶縁物含有量が6%超と多い（過剰）ため絶縁性被覆層の形成が充分で高周波特性は優れるものの、その反面磁心中の鉄粉の割合が不足するために実効透磁率、磁束密度および抗折力が劣り、所望の品質に達しない。これに対して、特に好ましいのは絶縁物が5%（鉄粉95%）であるが、絶縁物が1～6%の範囲内にある試料4～試料16は磁気特性、強度とも所望の品質を充分満たしている。従って試料4～試料16がこの発明の実施例であり、試料1～試料3は絶縁物（無機絶縁物と有機絶縁物の和）の配合量が1%未満で高周波特性が劣ること；試料17～試料19は絶縁物が6%超と過剰で透磁率、磁束密度、強度ともに劣ることにより、何れもこの発明の範囲外とされる。次の試料20～試料23はいわゆる従来型の圧粉磁心で、絶縁物の配合量は実施例中最適な5%を採択し、原料粉を混合したまま融合処理を省いて圧縮成形した比較例である。

【0020】ここで、この発明の特徴とする融合処理に

についての検討に先立ち、他の要因に関する検討結果を説明する。先ず無機絶縁物の種類については、配合量が等しく4.5%の試料6～試料15を見ると、各特性とも炭酸カルシウムの数値が幾分高いものの全体として有意差は認められない。これは絶縁物配合量が過剰の試料18, 19からも同様である。

【0021】次に鉄粉へのリン酸被膜処理の効果については、融合処理をしない比較例では一応有効と思われるが、融合処理を施した場合は試料7～試料12を見ると相反するデータがあり、明らかに有効とは言い難い。その理由は、融合処理の過程で受ける強力な摩擦および圧縮・剪断作用によってリン酸被膜の効果が減殺されるためと考えられる。なおリン酸被膜処理を施した鉄粉は流動性などの粉末特性が改善されるためか、融合処理の有無に拘らず抗折力が高めになっている。

【0022】成形時の成形圧力については、その影響を受けるのは主に磁束密度と抗折力の筈であり、事実低圧で成形した試料6～試料8および試料13～試料15と高圧で成形した試料9～試料12とを比べると磁束密

*度、抗折力ともに有意である。また抗折力については、成形後の加熱温度との相関傾向も見受けられる。

【0023】成形後の加熱（固化）処理温度の影響については、この加熱は一般に圧粉体を強化させ、また成形圧力によって圧粉体に残留する歪みを除去する。圧粉磁心に残留する歪みは透磁率、保磁力、鉄損（ヒステリシス損失）などの構造敏感性の磁気特性を阻害するので、加熱によって歪みが消失すれば、これらの特性が向上する筈である。しかしその一方で、鉄粉の表面に形成された被覆層の絶縁成分が加熱によって鉄粉内部に拡散すると、本来の絶縁効果が損われ、結果として磁気特性を劣化させる。加熱温度180℃の試料7, 9; 350℃の試料6, 10; 500℃の試料11, 12が示すように、高温になるほど抗折力と透磁率は向上するものの高周波特性は劣化の傾向にあるのは、歪みの消失と拡散の進行という効果が相反する作用のためと考えられる。

【0024】

【表1】

試験片の作製条件

試料 No.	原料粉配合組成（体積％）						融合 処理	成形圧 MPa	加熱 ℃	備 考
	還元鉄粉	滑石	Al ₂ O ₃	SiO ₂	CaCO ₃	樹脂				
1	99.8	—	—	—	—	0.2	○	588	180	範囲外
2	99.3	0.2	—	—	—	0.5	○	588	180	範囲外
3	99.3	—	0.2	—	—	*0.5	○	588	350	範囲外
4	99.0	0.5	—	—	—	0.5	○	588	180	本発明
5	97.0	2.5	—	—	—	0.5	○	588	180	本発明
6	95.0	—	4.5	—	—	*0.5	○	588	350	本発明
7	95.0	4.5	—	—	—	0.5	○	588	180	本発明
8	☆95.0	4.5	—	—	—	0.5	○	588	180	本発明
9	☆95.0	4.5	—	—	—	0.5	○	980	180	本発明
10	95.0	—	4.5	—	—	*0.5	○	980	350	本発明
11	☆95.0	4.5	—	—	—	0.5	○	980	500	本発明
12	95.0	—	4.5	—	—	*0.5	○	980	500	本発明
13	95.0	—	—	4.5	—	*0.5	○	588	350	本発明
14	95.0	—	—	—	4.5	*0.5	○	588	350	本発明
15	95.0	—	1.5	1.5	1.5	*0.5	○	588	350	本発明
16	94.0	—	1.5	1.5	2.5	*0.5	○	588	350	本発明
17	93.0	—	2.0	2.0	2.5	*0.5	○	588	350	範囲外
18	92.5	7.0	—	—	—	0.5	○	588	180	範囲外
19	92.5	—	7.0	—	—	*0.5	○	588	350	範囲外
20	95.0	4.5	—	—	—	0.5	—	588	180	比較例
21	☆95.0	4.5	—	—	—	0.5	—	588	180	比較例
22	☆95.0	4.5	—	—	—	0.5	—	980	180	比較例
23	☆95.0	4.5	—	—	—	0.5	—	980	500	比較例

☆：鉄粉にリン酸被膜処理

*：ポリフェニレンサルファイド樹脂

（無印はフェノール樹脂）

【0025】

【表2】

磁気特性と強度の測定結果

試料 No.	配合の概要		融合 処理	実効透磁率 (μ)				磁束 密度	抗折力 MPa	備 考
	還元鉄粉	樹 脂		1kHz	400kHz	1MHz	1M/1k			
1	99.8	0.2	○	128	98	67	0.52	1.2	72	範囲外
2	99.3	0.5	○	120	96	76	0.63	1.2	79	範囲外
3	99.3	*0.5	○	117	93	82	0.70	1.2	85	範囲外
4	99.0	0.5	○	102	98	91	0.89	1.3	83	本発明
5	97.0	0.5	○	85	83	81	0.95	1.2	82	本発明
6	95.0	*0.5	○	75	74	71	0.95	0.9	69	本発明
7	95.0	0.5	○	74	73	71	0.96	0.9	69	本発明
8	☆ 95.0	0.5	○	74	74	73	0.99	0.9	71	本発明
9	☆ 95.0	0.5	○	80	78	77	0.96	1.1	79	本発明
10	95.0	*0.5	○	83	79	77	0.93	1.1	74	本発明
11	☆ 95.0	0.5	○	88	79	75	0.85	1.1	83	本発明
12	95.0	*0.5	○	85	80	77	0.90	1.1	81	本発明
13	95.0	*0.5	○	74	73	71	0.96	0.9	71	本発明
14	95.0	*0.5	○	79	78	77	0.97	1.1	79	本発明
15	95.0	*0.5	○	76	75	73	0.96	1.0	77	本発明
16	94.0	*0.5	○	67	67	63	0.94	0.8	65	本発明
17	93.0	*0.5	○	58	57	52	0.90	0.7	50	範囲外
18	92.5	0.5	○	52	51	50	0.96	0.7	49	範囲外
19	92.5	*0.5	○	54	52	51	0.94	0.7	45	範囲外
20	95.0	0.5	—	72	30	13	0.18	0.8	54	比較例
21	☆ 95.0	0.5	—	70	58	46	0.66	0.7	57	比較例
22	☆ 95.0	0.5	—	78	55	41	0.52	0.9	64	比較例
23	☆ 95.0	0.5	—	115	12	8	0.08	0.9	81	比較例

☆：鉄粉にリン酸被膜処理

*：ポリフェニレンサルファイド樹脂（無印はフェノール樹脂）

【0026】最後に、原料粉に対する融合処理の効果は、この処理を施した試料7～試料9および試料11を見ると、その全てが実効透磁率、高周波特性、磁束密度などの磁気特性、抗折力とも高周波用圧粉磁心として十分な品質を具え、中でも高周波特性は高压で成形した場合でも、僅かな低下に留まっている。その理由は、融合処理を経た被覆層は内部の鉄粉と強固に接合しているために、圧粉成形時の高い圧力や摩擦摩擦に充分耐え得ることにある。これに対して、融合処理を施さない試料20～試料23では、周波数が400kHzを超えると低圧で成形した場合でも実効透磁率が急激に減少し、高周波特性が著しく劣化している。これは被覆層と内部の鉄粉との接合が弱いため、成形時に受ける圧力や摩擦により被覆層の剥離、損傷が生じたことを意味する。そしてこの事実は、この発明の特徴とする融合処理の有効性を如実に示している。なお抗折力が成形圧力や加熱温度の上昇につれて向上しているのは、常識的に首肯されると

ころである。

【0027】

30 【発明の効果】従来、1MHzを超えるような高周波領域では、絶対値は低い（0.4前後）ものの磁束密度が安定しているフェライトコアが専ら用いられ、一方数十kHz程度までの領域では珪素鋼板積層品が用いられているが、その中間の領域用には適切な磁心材が無かった。例えばセンダストは、周波数1MHzでも磁束密度が安定してはいるがそのレベルはフェライトコアより若干高い程度のため、当初に述べた機器の小型化に対応することは出来ない。しかるに、この発明に係る圧粉磁心は周波数1MHzまで実効透磁率が殆ど低下しないため
40 高周波特性が優れ、且つ高い磁束密度（1.1前後）を示している。従ってこの発明は圧粉磁心の用途範囲を拡大するとともに、電気・電子機器の小型化への対応を可能にしたものである。